

COMPOSITE OF THE RECYCLED POLYPROPYLENE REINFORCED WITH
OIL PALM EMPTY FRUIT BUNCH

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ABSTRACT

This study is about the composite of recycled polypropylene reinforced with empty fruit bunch (EFB) fiber chopped into different length. The optimum weight percentage of EFB that gives higher strength and the effect of coupling agent on the composite are determined. The composite is prepared using EFB fiber with recycled polypropylene with weight percentage from 10 wt% to 50 wt%. Samples are prepared using extruder at 170 °C temperature with screw speed 70 rpm. The melt flow indexes (MFI) of the extruded samples are determined. Samples then injection moulded into dumb-bell specimen for testing and analysis. From the tensile testing, composite which has 30 wt% and 40 wt% of EFB shows optimum elongation modulus. The recycled polypropylene/ fiber composite with 30 wt% and 40 wt% EFB are prepared by adding coupling agent (MAPP) with fiber and MAPP ratio (10:1). Stress-strain properties of the composite are analyzed. The reinforcing property of the recycled polypropylene/ EFB fiber composite with and without coupling agent is compared.

ABSTRAK

Kajian dijalankan atas komposit dengan menggunakan polypropylene yang dikitar semula bersama serat tandan kelapa sawit. Objektif kajian ini adalah untuk mengenal pasti peratus berat yang optimum tandan kelapa sawit dan pengaruh “coupling agent” (MAPP) yang memberi hasil komposit yang kuat. Komposit ini disediakan dengan menggunakan serat tandan kelapa sawit dan polypropylene yang dikitar semula pada peratusan berat 10% hingga 50%. Sampel disediakan dengan menggunakan plastik extruder pada suhu 170°C dan kelajuan 70 rpm. Kemudian sampel-sampel tersebut telah ditentukan akan index “melt flow”nya. Selepas itu, sampel tersebut telah melalui “injection moulding” proses dimana sampel dibentuk mengikut acuan yang dikehendaki untuk menguji kekuatan Tensile komposit. Hasil kajian menunjukkan berat serat kelapa sawit 30 % dan 40% memberi keputusan yang optimum. Polypropylene yang dikitar semula / serat tandan kelapa sawit komposit dengan peratusan berat 30% dan 40% termasuk MAPP (nisbah 10:1=serat: MAPP) telah disediakan. Ketegangan dan stres sampel tersebut telah dianalisis. Ciri-ciri komposit polypropylene yang dikitar semula/ serat tandan kelapa sawit bersama MAPP dan tanpa MAPP telah dibandingkan.

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LIST OF ABBREVIATION

RPP	- Recycled Polypropylene
MAPP	- maleic anhydride-grafted polypropylene
FTIR	- Fourier Transform Infrared Spectroscopy
EFB	- Empty Fruit Bunch
PP	- Polypropylene
OPEFB	- Oil Palm Empty Fruit Bunch
PVC/ENR	- Poly(Vinyl Chloride)/Epoxidized Natural Rubber
GF	- Glass Fiber
TPM	-3-(Trimethoxysilyl)-Propylmethacrylate
PU	- Polyurethane
MDI	- Diphenyl Methane Diisocyanate
PEG	- Polyethylene Glycol
HMDI	- Hexamethylene Diisocyanate
TDI	- Toluene Diisocyanate

CHAPTER 1

INTRODUCTION

1.1 Background Study

Composite is combination of two or more materials (reinforcing elements, fillers, and composite matrix binder), differing in form or composition on a macro scale. The constituents retain their identities, that is, they do not dissolve or merge completely into one another although they act in concert. Normally, the components can be physically identified and exhibit an interface between one another. The compounds of the composite consist of two main phases; matrix and fillers. The matrix is a continuous phase which bounds the filler and forms composite. (William D. Callister, Jr et al.,2008).

Recently the Interest in ecology-friendly or "green" polymer composites is growing due to concerns with increasing carbon emission and the limited nature of petroleum and natural gas resources. Hence, research and engineering interest has been shifting from traditional monolithic materials to fiber reinforced polymer-based materials due to their unique advantages of high strength to weight ratio, non-corrosive property and high fracture toughness. (Hoi-yan Cheung et al.,2009).

Fiber reinforced polymers, specifically with glass fibers, have gained importance in technical applications such as the automotive sector, where high mechanical properties and dimensional stability have to be combined with low

weight. Unfortunately, these fibers have serious drawbacks such as (i) non-renewable, (ii) non-recyclable, (iii) high energy consumption in the manufacturing process, (iv) health risk when inhaled and (v) non-biodegradable. (Hoi-yan Cheung et al.,2009). During the last few years many efforts have been made to investigate the suitability of natural fibers as a reinforcing component for thermoplastic and injection moldable materials because of their low density and ecological advantage. (Martina Wollerndorfer et al.,1997).

They open up further possibilities in waste management as they are biodegradable and therefore can lead to highly functional composite materials if used in combination with recycled thermoplastic polymers. (Martina Wollerndorfer et al., 1997). Since the polypropylenes are used widely in the production of car bumpers, the automotive industries are looking ways to recycle the polypropylene and reduce the other polymers usage. (Agnes F. Martins et al., 1999). Apart of that the usage of recyclates are being encouraged to reduce carbon dioxide accumulation in the atmosphere and dependence on petroleum-derived materials. These issues lead to the research using recyclates such as recycled polypropylene.

Although the bio-filler filled composite materials have several advantages, it also has its own disadvantages that are the Hydrophilic character of the fiber and the hydro phobic character of the polymer. These lead to the low compability which causes low mechanical properties. To improve the bonding between the fiber and the matrix the role of coupling agent is very important. The suitable coupling agent for matrix polypropylene is maleic anhydride-grafted polypropylene (MAPP). MAPP helps to enhance the interfacial adhesion which improves the mechanical and thermal stability of the composite.

1.2 Problem statement

The main purpose of this study is to produce composite of recycled polypropylene reinforced with oil palm empty fruit bunch fiber which is something new in the polymer composite area. There are many researches being done using glass fibers with matrix as polypropylene and polyethylene in the composite developments. But this will be the first time where recycled polypropylene is used to produce composite with empty fruit bunch fiber as fillers.

There are three major problems in the considerations in carrying out this project. The main consideration is actually the cost of raw materials. The raw material that we are talking here is petroleum. Production of composite using polypropylene is more expensive compare to recycled polypropylene. Therefore, composite of recycled polypropylene reinforced with oil palm empty fruit bunch fiber is opted. In that case the recycled polypropylene from car parts recycling industry and empty fruit bunch from the oil palm industry plays important role as a continuous source of raw material for this process. It can also result in substantial reductions in non-renewable energy consumption.

Secondly, environmental consideration will be the other problem to be resolved. Disposal of plastic products such as bottles, plastic bags and food wrappers to landfill is generally practiced. Large quantity of disposal causes the landfill-space crisis is especially problematic in cities, where inner-city trash dumps are often filled to capacity, and surrounding communities are unwilling to allow new landfills to come to their neighborhoods. Hence, fiber incorporated into plastics such as recycled polypropylene materials can reduce the overall usage of non-biodegradable derived materials. These can decrease the release of sequestered carbon, able to increase the use of renewable carbon-neutral materials and reduces the environmental impact of non-biodegradable materials.

In addition to environmental factors, natural fibers or "bio-fibers" have many advantages over traditional polymer fillers such as glass fiber. These include low

cost, low energy consumption, non-abrasive nature, safety in handling, low density, potentially higher volume fraction, superior specific properties, etc. Bio-fibers such as hemp, jute, kenaf, sisal, bamboo and empty fruit bunch can potentially replace glass fiber to reinforce polymeric resins.

1.3 Objective

The objectives of the research are as below;

- 1) Increase the strength of recycled polypropylene by reinforcing EFB.
- 2) Test the effect of coupling agent to optimize the strength of material.
- 3) To analyze and test the performance of reinforced recycled polypropylene with EFB.

1.4 Scope

Based on the objectives, the main scope of this project is to study the processing of recycled polypropylene reinforced with EFB fiber composite by extrusion and injection molding. The scope of study includes the testing of the composite of its mechanical performance by performing tensile test, where the tensile strength, break strain and elongation modulus/ young's modulus is calculated. In this study the effect of coupling agent MAPP (maleic anhydride-grafted polypropylene) on the mechanical performance of the composite also will be conducted. In addition to that, Fourier Transform Infrared Spectroscopy (FTIR) and the density of the composite also studied.

CHAPTER 2

LITERATURE REVIEW

In past few years, the application and development of environmentally friendly materials such as bio-filler reinforced polymer composites have been widely studied in line with the rising environmental awareness thorough out the world. Recently, conservation of forests and optimal utilization of agricultural and other renewable resources like solar and wind energies, and recently, tidal energy have become important topics worldwide. In such concern, the use of renewable resources such as plant and animal based fibre-reinforce polymeric composites, has been becoming an important design criterion for designing and manufacturing components for all industrial products. Research on biodegradable polymeric composites, can contribute for green and safe environment to some extent. In the biomedical and bioengineered field, the use of natural fibre mixed with biodegradable and bioresorbable polymers can produce joints and bone fixtures to alleviate pain for patients. (Hoi-yan Cheung et al.,2009).

Another study conducted in Saudi Arabia where date palm leaves were compounded with polypropylene (PP) and UV stabilizers to form composite materials. The stability of the composites in natural weathering conditions of Saudi Arabia and in accelerated weathering conditions was investigated. The composites were found to be much more stable than PP under the severe natural weathering conditions of Saudi Arabia and in accelerated weathering trials. Compatibilized samples are generally less stable than uncompatibilized ones as a result of the lower stability of the maleated polypropylene. Irgastab and Tinuvin are found to be

efficient stabilizers for PP/cellulose fibre composites. In addition to enhanced stability imparted by the presence of the fibres in the composites, enhanced interfacial adhesion resulting from oxidation of the polymer matrix can be the source of retention of mechanical strength. (B.F.Abu-Sharkh et al.,2003)

From a study conducted in Austria it is known that, natural fibres are of basic interest since they not only have the functional capability to substitute the widely used glass fibers but they also have advantages from the point of view of weight and fibre–matrix adhesion, specifically with polar matrix materials. They have good possibilities in waste management due to their biodegradability. The influence of plant fibres such as flax, jute, ramie, oil palm fibres and fibres made from regenerated cellulose on the mechanical properties of biodegradable polymers was investigated using thermoplastics like polyesters, polysaccharides and blends of thermoplastic starch. In this study, the composites were produced by extrusion compounding with a co-rotating twin screw extruder. The pellets obtained were further processed into tensile test bars by injection moulding. Depending on the kind of polymer, a fibre content of 20–35% achieved. Generally a considerable tensile strength improvement of polyesters could not be observed. However the chemical similarity of polysaccharides and plant fibres, which consist mainly of cellulose, resulted in an increased tensile strength of the reinforced polymers. For reinforced thermoplastic wheat starch, it was four times better (37 N/mm²) than without fibres. The reinforcement of cellulose diacetate and starch blends caused a stress increase of 52% (55 N/mm²) and 64% (25 N/mm²), respectively. (Martina Wollerdorfer et al.,1997).

EFB and sisal fibers are used to reinforce to improve the strength of the rubber in a study conducted in India, Kerala state where the natural rubber is reinforced with untreated sisal and oil palm fibers chopped to different fiber lengths. The effects of concentration and modification of fiber surface in sisal/oil palm hybrid fiber reinforced rubber composites have been studied. Increasing the concentration of fibers resulted in reduction of tensile strength and tear strength, but increased modulus of the composites. Composites were prepared using fibers treated with varying concentrations of sodium hydroxide solution and for different time intervals.

The vulcanisation parameters, process ability characteristics, and stress–strain properties of these composites were analysed. The rubber/fiber interface was improved by the addition of a resorcinol-hexamethylene tetramine bonding system. The reinforcing property of the alkali treated fiber was compared with that of untreated fiber. The extent of fiber alignment and strength of fiber-rubber interface adhesion were analysed from the anisotropic swelling measurements. (Maya Jacob et al.,2003).

In another study using natural rubber, the effect of irradiation on the tensile properties of oil palm empty fruit bunch (OPEFB) fiber reinforced poly(vinyl chloride)/epoxidized natural rubber (PVC/ENR) blends were studied. The composites were prepared by mixing the fiber and the PVC/ENR blend using HAAKE Rheomixer at 150°C. The composites were then irradiated by using a 3.0 MeV electron beam machine at doses ranging from 0 to 100 kGy in air and room temperature. The tensile strength, Young's modulus, elongation at break and gel fraction of the composites were measured. Comparative studies were also made by using poly(methyl acrylate) grafted OPEFB fiber in the similar blend system. An increase in tensile strength, Young's modulus and gel fraction, with a concurrent reduction in the elongation at break (Eb) of the PVC/ENR/OPEFB composites was observed upon electron beam irradiation. Studies revealed that grafting of the OPEFB fiber with methyl acrylate did not cause appreciable effect to the tensile properties and gel fraction of the composites upon irradiation. The morphology of fractured surfaces of the composites, examined by a scanning electron microscope showed an improvement in the adhesion between the fiber and the matrix was achieved upon grafting of the fiber with methyl acrylate. (Chantara Thevy Ratnam et al.,2007)

There are a lot of researches done using hybrid fillers to produce composite. One of the studies are done at local university on polypropylene reinforced with oil palm empty fruit bunch (EFB) and glass fiber (GF).Both flexural and tensile modulus have been improved with increasing level of overall fiber content loading. The effect of the coupling agent also tested here and the result shows that maleic anhydride-grafted polypropylene(MAPP) and 3-(trimethoxysilyl)-

propylmethacrylate (TPM) had imparted considerable improvements in flexural and tensile properties. (H.D.Rozman et al.,2001).

Another study conducted using EFB fiber by the same university but this time the use polyurethane (PU) as the matrix and produced polyurethane (PU) - oil palm empty fruit bunch (EFB) composites. The PU matrix employed consisted of diphenyl methane diisocyanate (MDI) and polyethylene glycol (PEG) with a molecular weight of 200. EFB fibres were used in the form of mats. The fibres were treated with two types of isocyanate: hexamethylene diisocyanate (HMDI) and toluene diisocyanate (TDI). In general, the employment of EFB in mat form has produced PUEFB composites with acceptable properties. The properties of the composites were believed to be predominantly influenced by the type of bonding produced. In general, the composites with isocyanate treated fibres showed superior tensile and flexural properties than those without treatment. These were believed to be attributed to the additional reaction sites in the form of urethane functional groups produced as the result of NCO reactions with OH of EFB prior to subsequent interaction with PU/PEG mixtures. The relatively superior properties of composites with HMDI treated fibres over those treated with TDI were probably due to the longer chain of the former, which render it more accessible to the reaction with OH of PEG and also to its increased capability in absorbing more energy transferred from the matrix. (H.D.Rozman et al.,2001).

In this work, done in Bangladesh on 2009, palm and coir fiber reinforced polypropylene bio-composites were manufactured using a single extruder and injection molding machine. Raw palm and coir were chemically treated with benzene diazonium salt to increase their compatibility with the polypropylene matrix. Both raw and treated palm and coir fiber at five level of fiber loading (15, 20, 25, 30 and 35 wt.%) was utilized during composite manufacturing. Micro structural analysis and mechanical tests were conducted. Comparison has been made between the properties of the palm and coir fiber composites. Treated fiber reinforced specimens yielded better mechanical properties compared to the raw composites, while coir fiber composites had better mechanical properties than palm fiber ones. Based on

fiber loading, 30% fiber reinforced composites had the optimum set of mechanical properties. (Md. Mominul Haque et al.,2009).

In this study different approach is used using EFB where the cellulose was derived from oil palm empty fruit bunch fiber (EFBF) by standard ASTM D1104 method. The cellulose and EFB fibers were blended in different ratios up to 50-wt.% with polypropylene (PP) using Brabender twin-screw compounder. Effects of cellulose and EFB fibers on the mechanical properties of PP were investigated. Studies on the morphological properties and the influence of fiber loading on the properties of PP-cellulose and PP-EFBF composites were also conducted. The PP-cellulose composite gave better results in comparison with PP-EFBF composite. The changes in mechanical and morphological properties with different cellulose and fiber loading were discussed. (M. Khalid et al.,2007).

The coupling agent improves the mechanical properties of the polymer composite. This can be seen in this study where the effect of processing temperature on the interfacial adhesion, mechanical properties and thermal stability of bio-flour-filled, polypropylene (PP) composites was examined as a function of five different maleic anhydride-grafted PP (MAPP) types. To investigate the effect on the interfacial adhesion of the composites, the five MAPP types were subjected to characterization tests. The MAPP-treated composites with sufficient molecular weight and maleic anhydride (MA) graft (%) showed improved mechanical and thermal stability. The enhanced interfacial adhesion and mechanical and thermal stability of the MAPP-treated composites was strongly dependent on the amount of MA graft (%) and the MAPP molecular weight. The morphological properties of the MAPP-treated composites showed strong bonding and a paucity of pulled-out traces from the matrix in the two phases. In addition, the improved interfacial adhesion of the MAPP-treated composites was confirmed by spectral analysis of the chemical structure using attenuated total reflectance (FTIR-ATR). The crystallinity of PP, MAPP, MAPP-treated composites and non-treated composites was investigated using wide-angle X-ray scattering (WAXS) and differential scanning calorimetry (DSC). (Hee-Soo Kim et al.,2007).

From the literature review, clearly seen that there are limited almost no work is done using recycled polypropylene. The studies using EFB fiber as reinforcing agent shows very good results. It also shows that the effective coupling agent to improve the polypropylene composite is maleic anhydride (MA) graft. Since oil palm is a major plantation in Malaysia and EFB fiber is available free of charge, it is a perfect material to use as the reinforcing agent to improve the mechanical properties of the recycled polypropylene with adding MAPP as coupling agent.

CHAPTER 3

METHODOLOGY

3.1 Introduction

Experiment method is the main method used in this research. The process used to produce the recycled polypropylene- oil palm EFB fiber composite is twin screw extrusion. There are four major parts in this experiment. First, extrusion process of recycled polypropylene-oil palm EFB fiber various weight percentage of EFB fiber and secondly the measurement of melt flow index (MFI) of produced composite, using melt flow index (MFI) machine, thirdly injection molding the samples into dumbbell shape for tensile testing, fourthly doing mechanical testing and analysis of the produced composite and finally repeating the same process from first to fourth but this time adding the coupling agent, MAPP (maleic anhydride-grafted polypropylene).

The main part of this research is to produce recycled polymer composite reinforced with EFB fiber and the effect of coupling agent, MAPP on the mechanical properties of the composite produced.

3.2 Material

Recycled polypropylene (PP Black copo) mainly from car bumper and vehicle battery case with melt flow index (MFI) 5g/min to 10 g/min. (maleic anhydride-grafted polypropylene MAPP commercial name is polybond 3200 with density is 0.941 g/cm³ was supplied by Petronas Polymers Marketing and Trading Division Malaysia. Empty fruit bunch fiber (EFBF) was obtained from FELDA Palm Oil refinery in Pahang.



Figure 3.1: Recycled polypropylene (PP Black copo)

3.3 Equipment

3.3.1 Extruder

Extruder is a machine where the extrusion process takes place. In the extrusion process the polymer is forced through a die orifice by a compressive force that is applied to a ram; the extruded piece that emerges has the desired shape and a reduced cross section area. The polymer is propelled continuously along a screw through a region of high temperature and pressure where it is melted and compacted through a die into final shape. A wide variety of shapes can be made by extrusion, including rods, channels and other structural shapes, tubing and hose, sheeting up to several feet wide and one-quarter inch or more thick, and film of similar width down to a few thousandths of an inch in thickness.

The screw of an extruder is divided into several sections, each with a specific purpose. The feed section picks up the finely divided polymer from a hopper and propels it into the main part of the extruder. In the compression section, the loosely packed feed is compacted, melted and formed into a continuous stream of molten plastic. Some external heat must be applied, but much is generated by friction. The metering section contributes to uniform flow rate, required to produce uniform dimensions in the finished product, and builds up sufficient pressure in the polymer melt to force the extruder and out of the die. Since viscous polymer melts can be mixed only by the application shearing forces (their viscosity is too high to allow turbulence or diffusion to contribute appreciably to mixing), an additional working section may be needed before the die.

Modern trends in extruder usage include the twin-screw or multiple-screw extruder, in which two screws turn side by side in opposite directions, providing more working of the melt, and the vented extruder, having an opening vent at some point along the screw that can be opened or led to vacuum to extract volatiles from the polymer melt.



Figure 3.2: Extruder

3.3.2 Injection Molder

Most thermoplastic materials are molded by the process of injection molding. Injection molding is a manufacturing process for producing parts from both thermoplastic and thermosetting plastic materials. Material is fed into a heated barrel, mixed, and forced into a mold cavity where it cools and hardens to the configuration of the mold cavity. After a product is designed, usually by an industrial designer or an engineer, molds are made by a mold maker (or toolmaker) from metal, usually either steel or aluminum, and precision-machined to form the features of the desired part. Injection molding is widely used for manufacturing a variety of parts, from the smallest component to entire body panels of cars.

The following are the characteristic of the injection molding process;

- Utilizes a ram or screw-type plunger to force molten plastic material into a mold cavity
- Produces a solid or open-ended shape that has conformed to the contour of the mold
- Uses thermoplastic or thermoset materials
- Produces a parting line, sprue, and gate marks
- Ejector pin marks are usually present

Injection molding is used to create many things such as wire spools, packaging, bottle caps, automotive dashboards, pocket combs, and most other plastic products available today. Injection molding is the most common method of part manufacturing. It is ideal for producing high volumes of the same object. Some advantages of injection molding are high production rates, repeatable high tolerances, the ability to use a wide range of materials, low labor cost, minimal scrap losses, and little need to finish parts after molding. Some disadvantages of this process are expensive equipment investment, potentially high running costs, and the need to design moldable parts.

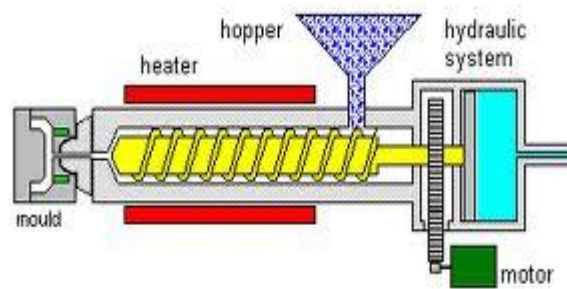


Figure 3.3: Injection Molder

3.3.3 Palletizer

Palletizer is used to palletize the extruded sample into small size as in the figure below.

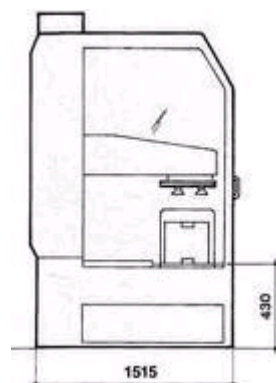


Figure 3.4: Palletizer

3.3.4 Grinder

Grinder is used to shred or chop the EFB fiber into small, about 8 to 10 mm in length.



Figure 3.5: Fiber Grinder

3.3.5 Melt Flow Index Machine

The melt flow index machine is used to obtain the MFI value of the samples. It's done after extrusion and before injection moulding. The purpose of the process is to identify the fluidity of the sample and to determine whether the sample is can be injection molded or otherwise.

3.3.6 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR), is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. FTIR technique has made dispersive infrared spectrometers all but obsolete (except sometimes in the near infrared) and opened up new applications of infrared spectroscopy.



Figure 3.6: FTIR

3.3.7 Pycnometer

A pycnometer or specific gravity bottle, is a device used to determine the density of a liquid. A pycnometer is usually made of glass, with a close-fitting ground glass stopper with a capillary tube through it, so that air bubbles may escape from the apparatus. This device enables a liquid's density to be measured accurately by reference to an appropriate working fluid, such as water or mercury, using an analytical balance.

If the flask is weighed empty, full of water, and full of a liquid whose specific gravity is desired, the specific gravity of the liquid can easily be calculated. The particle density of a powder, to which the usual method of weighing cannot be applied, can also be determined with a pycnometer. The powder is added to the pycnometer, which is then weighed, giving the weight of the powder sample. The pycnometer is then filled with a liquid of known density, in which the powder is completely insoluble. The weight of the displaced liquid can then be determined, and hence the specific gravity of the powder.



Figure 3.7: Pycnometer

3.3.8 Universal Tensile Machine

The universal tensile machine is used to obtain the Ultimate tensile strength (UTS), often shortened to tensile strength (TS) or ultimate strength,^{[1][2]} is the maximum stress that a material can withstand while being stretched or pulled before *necking*, which is when the specimen's cross-section starts to significantly contract. Tensile strength is opposite of compressive strength and the values can be quite different.

The UTS is usually found by performing a tensile test and recording the stress versus strain; the highest point of the stress-strain curve is the UTS. It is an intensive property; therefore its value does not depend on the size of the test specimen. However, it is dependent on other factors, such as the preparation of the specimen, the presence or otherwise of surface defects, and the temperature of the test environment and material.